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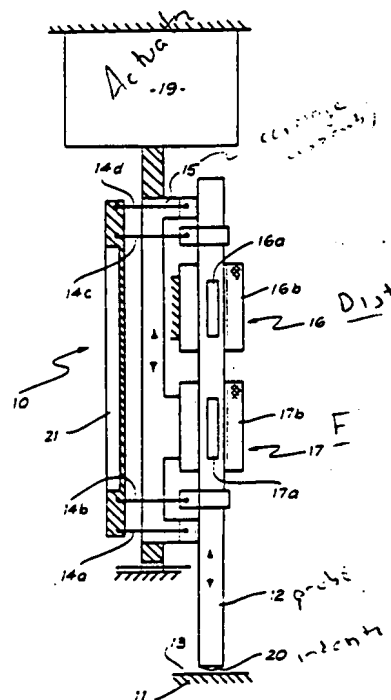
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With international search report.

(54) Title: PENETRATING MEASURING INSTRUMENT

(57) Abstract

A penetrating measuring apparatus (10) comprising: a stage (11) for mounting a sample to be tested, a probe (12) which extends at right angle to the stage (11) with an ultra-micro hardness indenter (20) in the end of the probe (12) nearest the stage (20), a carriage assembly (15) on which the probe (12) is mounted by elastic couplings (14a), (14b), (14c) and (14d) of known stiffness and an actuator (19) to drive the carriage (15) in a direction towards the stage (11). The probe is prevented from undergoing any but axial movements. First and Second measuring systems (16), (17) measure movement of the probe (12) relative to a known datum and the carriage (15) respectively. From the first measuring system (16) data is provided regarding actual penetration and the second measuring (17) system provides data indicating the force applied to the indenter. A control system for the inventive apparatus is also disclosed.



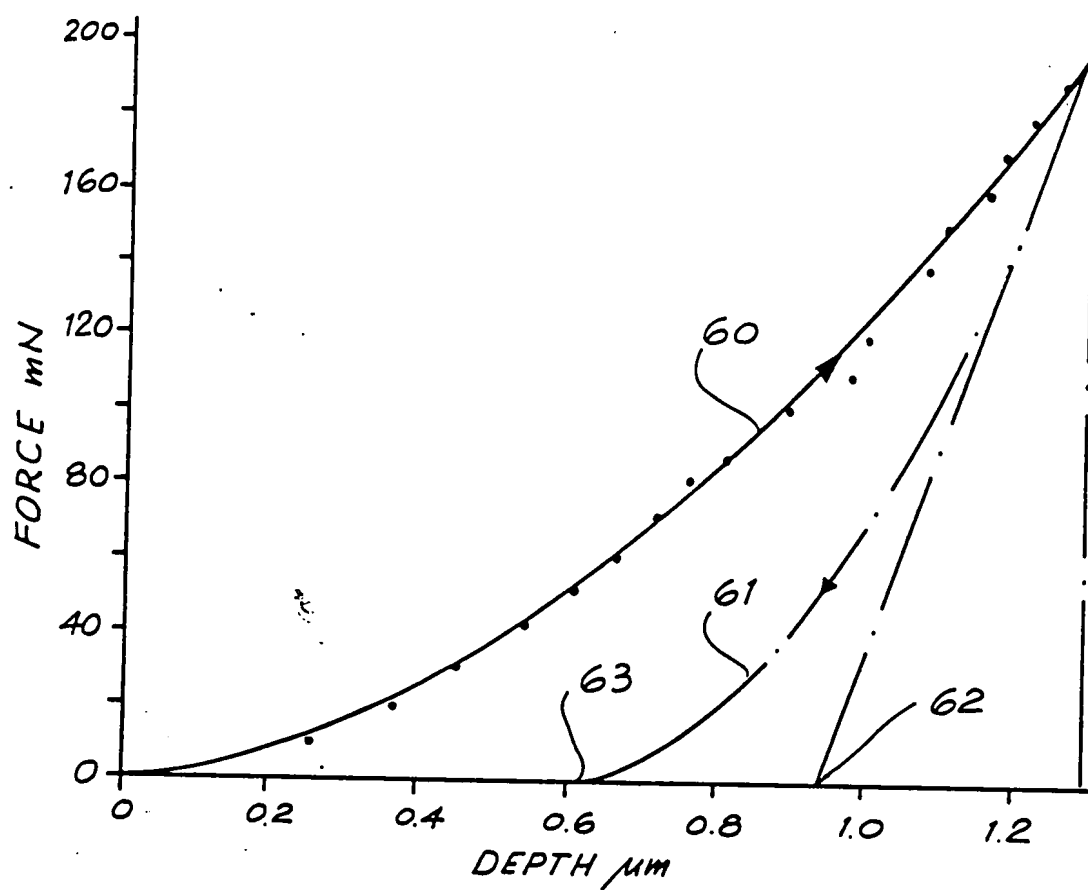
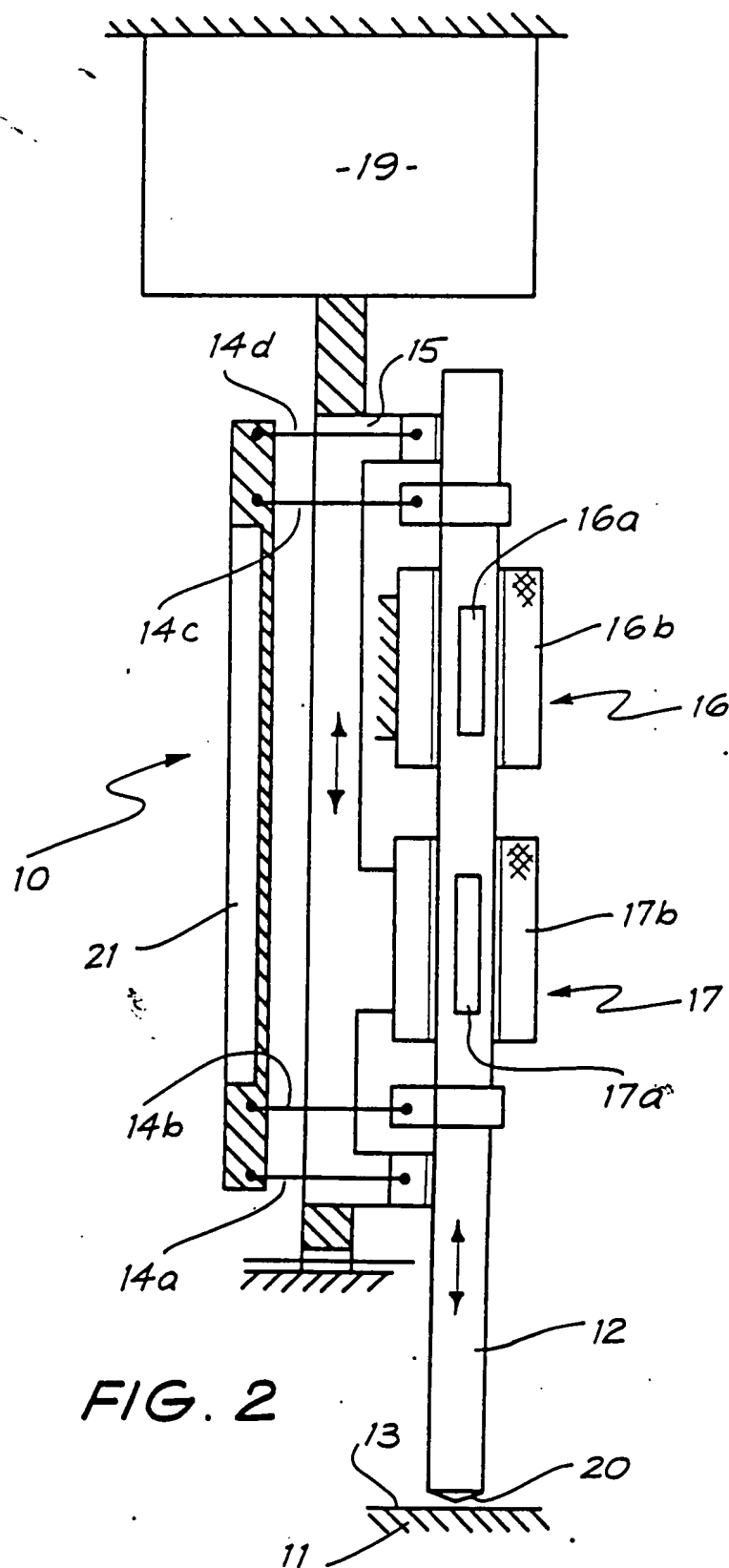
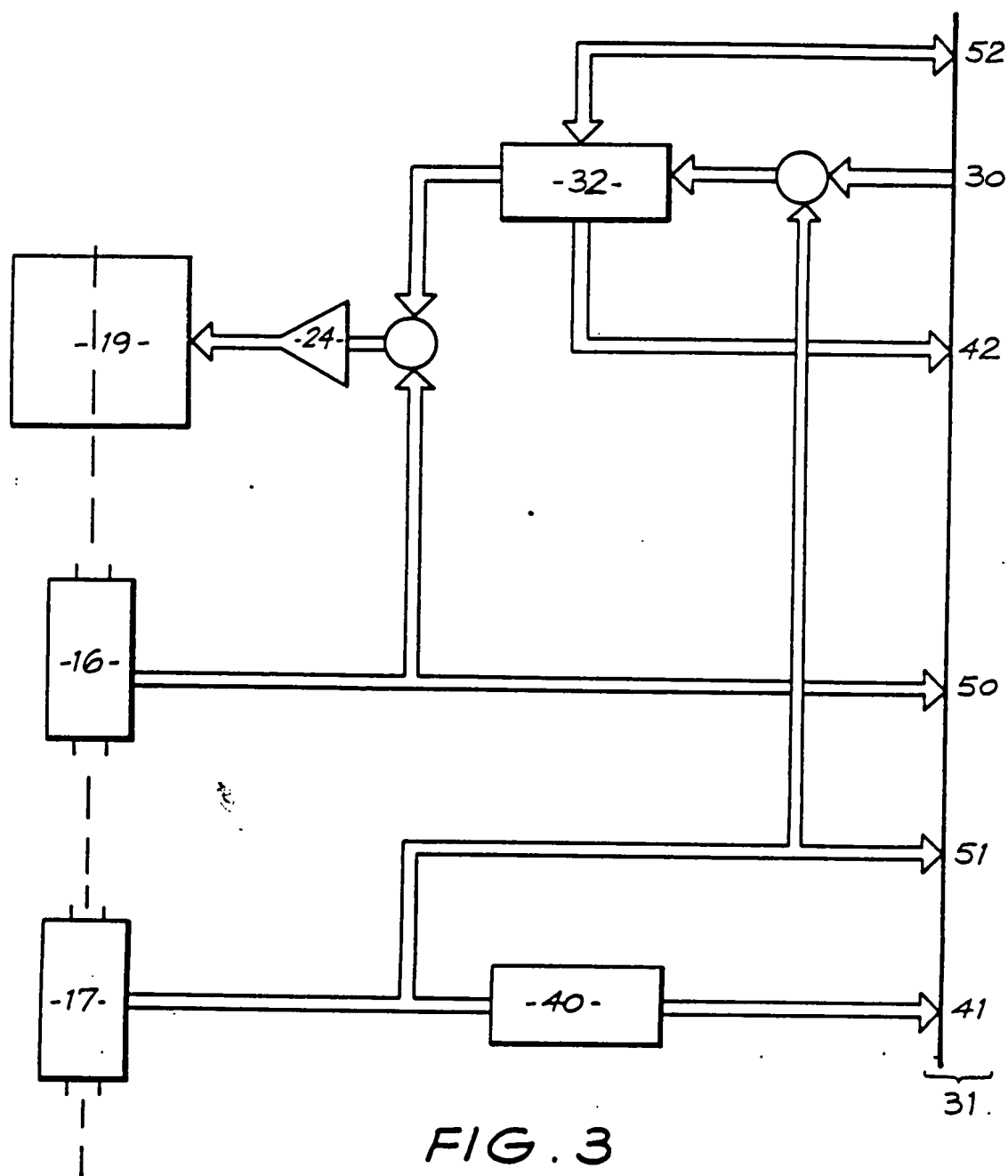


FIG. 1





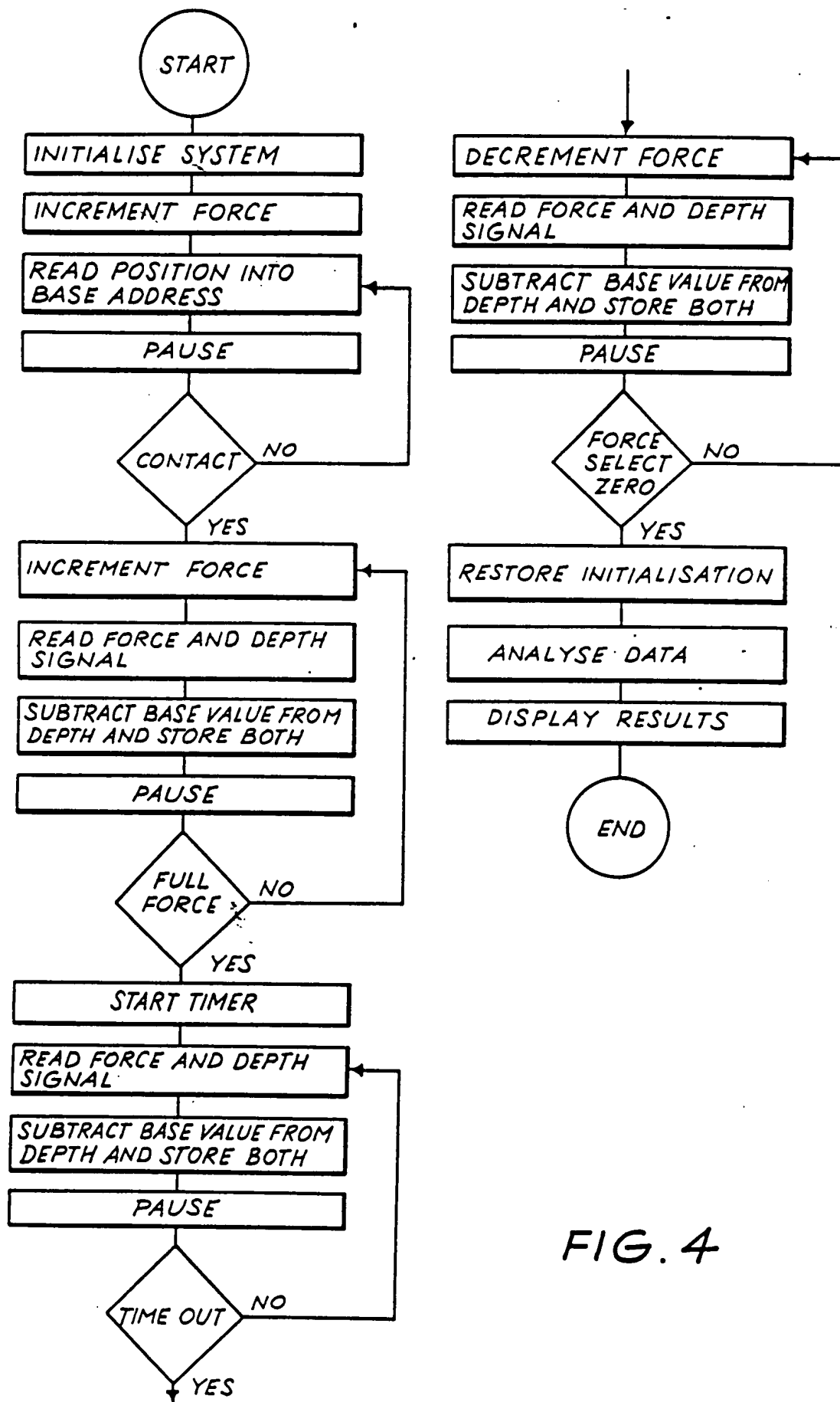
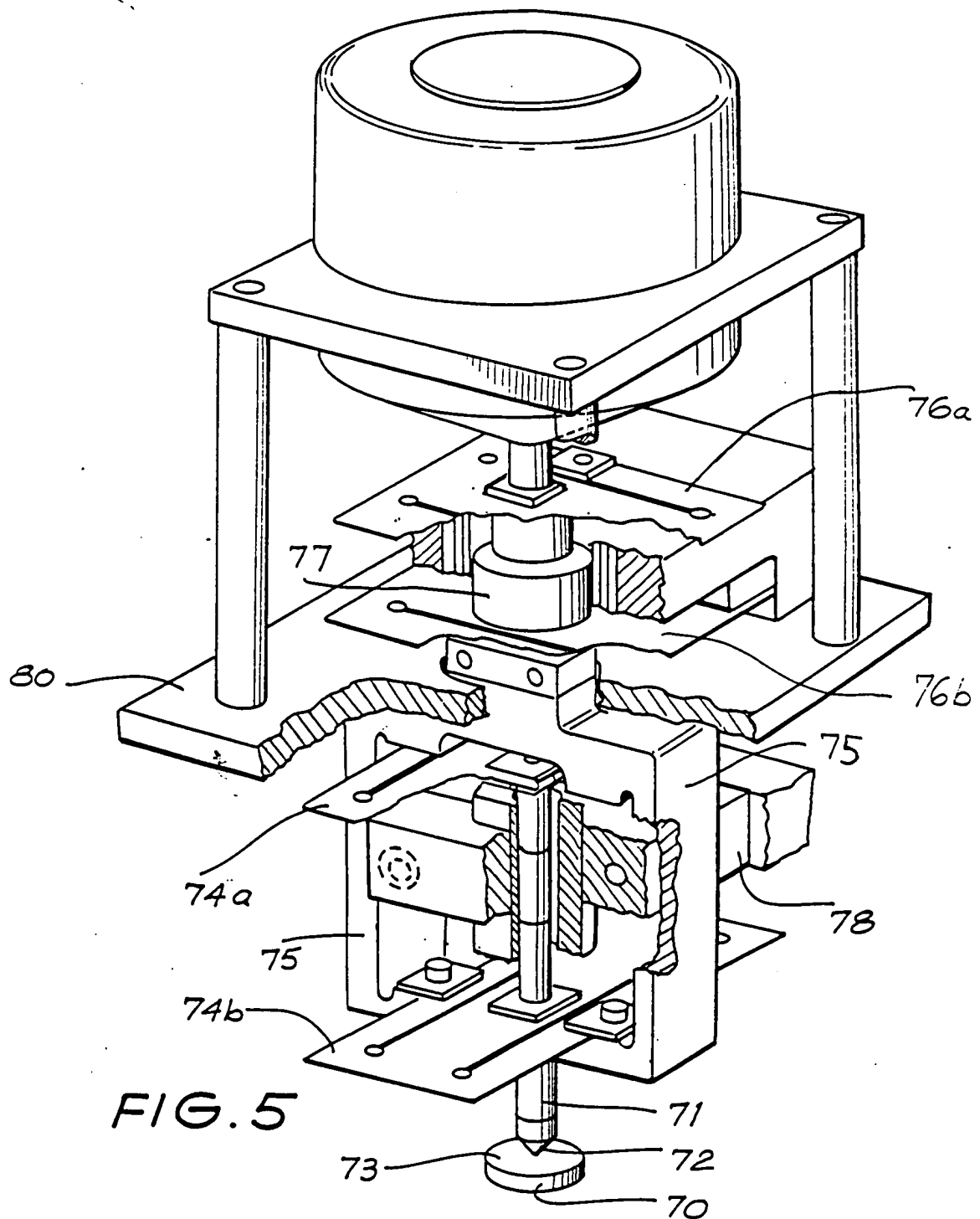


FIG. 4



INTERNATIONAL SEARCH REPORT

International Application No PCT/AU 87/00202

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| I. CLASSIFICATION OF SUBJECT MATTER : <small>use of classification symbols apply indicate all</small> According to International Patent Classification (IPC) or to both National Classification and IPC <div style="text-align: center; font-size: 1.2em;">Int. Cl.⁴ G01N 3/40, 3/42, 3/44</div> | | |
| II. FIELDS SEARCHED <div style="text-align: center; font-size: 0.8em;">Minimum Documentation Searched ?</div> <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> Classification System <div style="text-align: center; font-size: 1.2em;">IPC G01N 3/40, 3/42, 3/44</div> </div> <div style="width: 45%;"> Classification Symbols </div> </div> <div style="text-align: center; font-size: 0.8em; margin-top: 5px;"> Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched * </div> <div style="padding: 10px 0;"> AU: : IPC as above </div> | | |
| III. DOCUMENTS CONSIDERED TO BE RELEVANT * | | |
| Category * | Citation of Document, " with indication, where appropriate, of the relevant passages " | Relevant to Claim No. " |
| A P, A A A A A A | GB,B, 1566492 (ERNST) 30 April 1980 (30.04.80). Patent Abstracts of Japan, P537, page 65, JP,A, 61-193047 (NEC CORP) 27 August 1986 (27.08.86) Patent Abstracts of Japan, P174, page 37, JP,A, 57-184949 (SUMITOMO GOMU KOGYO KK) 13 November 1982 (13.11.82) Derwent Soviet Inventions Illustrated, Vol.U, No.50, Issued 17 January 1974 (17.01.74) SU,A, 373581 (TERNOVSKII) 8 June 1973 (08.06.73) Derwent Abstract Accession No. H 6087A/39, Class R16, SU,A, 579561 (NOVOSELCV) 28 November 1977 (28.11.77) Derwent Abstract Accession No. J 2946B/39, Class R16, SU,A, 638873 (MOSCENG-PHYS INST) 25 December 1978 (25.12.78) Derwent Abstract Accession No. 85/248313/40, Class S03, SU,A, 1147950 (BAIKOV METALLURG EX BAIK) 30 March 1985 (30.03.85) | |
| <div style="display: flex; justify-content: space-between; font-size: 0.8em;"> <div style="width: 48%;"> <p>* Special categories of cited documents: "</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 48%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p> </div> </div> | | |
| IV. CERTIFICATION | | |
| Date of the Actual Completion of the International Search <div style="text-align: center; font-size: 1.2em;">22 September 1987 (22.09.87)</div> | | Date of Mailing of this International Search Report <div style="text-align: center; font-size: 1.2em;">(09.10.87) 9 OCTOBER 1987</div> |
| International Searching Authority <div style="text-align: center; font-size: 1.2em;">Australian Patent Office</div> | | Signature of Authorized Officer <div style="text-align: center;"> <div style="display: inline-block; text-align: right; font-weight: bold;">R.A. MURRAY</div> </div> |

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON
INTERNATIONAL APPLICATION NO. PCT/AU 87/00202

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

| Patent Document Cited in Search Report | Patent Family Members |
|--|-----------------------|
| GB 1566492 | BR 7701217 |
| | FR 2343240 |
| | US 4107978 |
| | IT 1084904 |
| | CH 594879 |
| | IT 1085316 |
| | CH 590470 |
| | DE 2707873 |
| | JP 52117183 |
| | DE 2707939 |

END OF ANNEX

PENETRATING MEASURING INSTRUMENT

The present invention relates to a penetration measuring instrument for use in ultra-microindentation. The use of penetration devices to obtain information relating to the mechanical characteristics of a material being tested is very well known. Typically, an indenter is forced into a sample of a material and then extracted therefrom, leaving an indentation in the surface of the sample. [The depth of penetration, surface or projected area of the indentation and the force applied to the indenter provide information indicative of material properties such as hardness or modulus of elasticity.]

The modification of surface layers and the use of surface coatings to provide improved chemical, mechanical, electrical or decorative properties is rapidly increasing. Such modifications include very thin surface treatments such as vacuum deposited coatings ion-implanted surface layers and surface hardening. In order that the properties of such coatings and layers be better understood, testing apparatus has been developed to carry out ultra-microhardness testing.

Ultra-microindentation is a technique for obtaining information about the mechanical properties of a surface material from relationships between the depth of penetration of an indenter and resistance to its penetration. Ultra-microindentations are produced by pressing the indenter, which is usually a diamond pyramid, into the surface under the control of an ultra-microindenting system (UMIS).

To ensure that the probe of a UMIS does not cause cracking in the sample coating or surface layer and also to ensure that the characteristics of the coating are tested independently of the substrate characteristics, it has been recommend that the depth of penetration should be small in relation to the total layer or coating

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thickness. As, for example, the depth of penetration of implanted ions typically does not exceed $1\mu\text{m}$, it is desirable to be able to carry out microhardness testing using penetrations as little as $0.1\mu\text{m}$. If an indenter with Vickers type geometry is used to produce an indentation of $0.1\mu\text{m}$ depth, the diagonal width of the indentation will be of the order of $0.7\mu\text{m}$.

Measurement of impressions having widths less than about $1\mu\text{m}$ are impossible to perform without the aid of an electron microscope. Skilled addressees will be aware that electron microscopes are extremely expensive, both in initial purchase cost and operating cost. Consequently, there is a need for a penetration measuring instrument capable of use in ultra-microhardness testing which can provide data indicative of the depth of indentation without the need to perform further measurement. As an indenter will be of known geometry the width surface or projected area of an indenter can be desired if the depth of indentation is known. It has been shown that if a device can provide information indicative of the indenter position and the indenting force at intervals during the loading and unloading phases, the data thus generated is sufficient to allow deductions to be made about the dynamic and static resistance to abrasion, to local penetration and about the static and dynamic elastic properties of the new surface material. That is, it is possible to carry out the appropriate calculations of material properties without recourse to measurement by a microscope of any kind, be it optical, electron or otherwise.

In order for such a penetration measuring apparatus to be successful, the indenter motion should be produced by an actuator which is capable of producing smooth, vibration free motion at very low speed. The effects of any inertial forces which, for example, can arise from

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dead weight loading, must be avoided as far as possible. In the present invention the solution is to support the indenter by elastic coupling means on a carriage assembly and, as the carriage assembly is moved an indenting force
5 is transferred to the probe. In this way the deflection of the elastic coupling means is always just sufficient to balance the instantaneous resistance to penetration and, provided the indenting mechanism has low mass, and moves slowly, inertial forces are minimal.

10 In a preferred embodiment of the present invention, the indenter is smoothly brought to rest at a predetermined indenting force by progressively reducing the indenter velocity to zero as the force approaches its
15 preset final value. Feedback control of final indenter position ensures that the indenting force is maintained at the predetermined level regardless of creep. This system allows the indentation process to proceed smoothly to a preset maximum indenting force in one step, to approach
20 this value in a series of steps, to dwell at each step for a predetermined time, to retract the indenter either completely in one movement or stepwise. Furthermore it is possible to record force and depth at each step or,
alternatively, while the indenter is moving. With the aid of a digitally controlled co-ordinate stage, it is
25 possible to translate the test surface with the indenter in contact under constant or changing force.

According to the present invention, there is disclosed a penetration measuring apparatus comprising:

30 a stage for mounting a sample of a material to be tested thereon;

a probe having an end thereof proximal to said stage and having a longitudinal axis extending orthogonally of said stage;

an ultra-microhardness indenter located in said first
35 end of said probe;

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a carriage assembly to which said probe is connected by elastic connection means of known stiffness, said carriage assembly and connection means allowing axial movement of said probe but preventing movement of said probe in any other direction;

means to drive said carriage assembly towards and away from said stage;

means to measure the movement of said probe relative to a stationary datum; and

means to measure the deformation of said elastic connection means connecting said probe to said carriage.

The present invention will now be described by way of example with reference to the accompanying drawings, in which:-

Fig. 1 is a loading/unloading curve for an elastic/plastic material when tested by a device in accordance with the present invention;

Fig. 2 is a schematic cross-sectional view of a preferred embodiment of the present invention;

Fig. 3 is a schematic representation of the control system used to operate the present invention;

Fig. 4 is a control system flow chart; and

Fig. 5 is a further embodiment of the present invention.

Analysis of loading unloading curves may be understood in conjunction with the loading and unloading curves shown in Fig. 1. The loading curve is designated by numeral 60 and the unloading curve by numeral 61.

The surface of the material adjacent to the indentation is assumed to be depressed by an amount similar to that which would be produced by a circular flat frictionless punch with the same cross sectional area as that part of the indentation in contact with the indenter. This deflection is assumed to recover vertically when the indenting force is removed. At least

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one third of the unloading curve is usually found to be straight and its projection 62 on to the penetration axis represents this recovery.

The final intersection 63 of the unloading curve with the depth axis represents the unrecovered depth of the indentation and the difference between this point and the intersection of the projection of the initial part of the unloading curve represents recovery of the bottom of the indentation. It is therefore possible to separate the total penetration depth into components representing elastic and plastic deformations and to attribute mechanisms to each of them.

Conventional Vickers hardness is determined from the loaded area of contact between the indenter and the indentation which is calculated from the length of the diagonals of a recovered indentation. Since there is little elastic recovery of these diagonals they are also related to depth of penetration and consequently, after removal of contaminants such as the depression of the surface and any deflection of the UMIS structure, loaded area of contact may be calculated from depth. Although the triangular based indenter commonly used in depth measuring systems has no diagonals the same principles apply and contact area can also be calculated from depth of penetration. Hardness values obtained from the UMIS are therefore in principle equivalent to Vickers hardness values and where comparisons are possible have been found to have similar values.

The division of the unloading curve into components due to two different mechanisms allows the contribution to conventional hardness of the permanent plastic deformation and the non-permanent elastic deformation of the material surrounding the indentation to be investigated. The ratio of these two contributions varies for different materials and therefore comparison of hardness values for different

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materials may be influenced by this ratio, and much investigation remains to be done. One method of discovering the simple hardness and modulus values of a sample using a UMIS, a single force, with any value within a selected range, and a dwell time between the achievement of full force and its removal are specified. The depth of penetration is measured and recorded after the specified dwell. The force is then reduced to a lower value than the specified value and the partially recovered depth is measured and recorded, again after the specified dwell. Ultra-microhardness Hu and elastic modulus E are calculated from these measurements using

$$H_u = F/k_1 (R_1 - k_2(R_1 - R_2))^2$$

$$E/(1-\nu^2) = F(k_3(R_1 - k_2(R_1 - R_2)) (R_1 - R_2))$$

in which F is the indenting force, R_1 and R_2 are the depth measurements, k_1 and k_3 are constants dependent on the type of indenter used and k_2 a constant dependant on the amount by which the force is reduced. Poission's ratio can be assumed to be approximately 0.3.

These aspect of hardness are amenable to study with the UMIS and represent one of its applications.

Fig. 2 illustrates a penetration measuring device 10, which embodies the present invention. The embodiment 10 comprises a stage 11 for mounting a sample (not shown) thereon, a probe 12 which extends axially in a direction orthagonal to the planar top surface 13 of the stage 11 and which is connected by elastic connection means 14a, 14b, 14c and 14d to a carriage 15 and an actuator 19 to drive the carriage 15 towards the stage 11. Furthermore, there is a first measurement device 16 which measures the movement of the probe 12 with respect to a datum and a second measuring device 17 which measures movement of the

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probe 12 relative to the carriage 15 and, therefore, any deformation of the elastic connection means 14a, 14b, 14c and 14d. An ultra-microindenter 20 is located on the end of the probe 12 which is proximal to the surface 13 of the stage 11.

In order to minimize the inertial effects referred to above and to allow accurate measurements of the movement of the components of the embodiment of Fig. 2, it is preferable that the actuator 19 be capable of producing smooth, vibration free motion of the probe 12 at low velocity. Many types of drives suitable for this purpose will be known to skilled addressees but the present applicant has found linear electromagnetic actuators and piezoelectric actuators to be preferable, however, electromagnetic shakers also provide adequate performance. Stiffness within the actuator is of importance in order to maximise control over indenting velocity as the resistance of penetration increases. It is noted that piezoelectric actuators are stiff but are also relatively expensive whilst linear electromagnetic actuators are relatively inexpensive and can be stiffened with positional feedback.

In the embodiment illustrated in Fig. 2, the elastic couplings 14a, 14b, 14c and 14d, take the form of leaf-springs. Such leaf-springs have relatively low and determinable stiffness in the indenting direction but have relatively high stiffness in directions transverse to the indenting direction. It will be clear to skilled addressees that in view of the small size of the indentations made by embodiments of the present invention, the probe must be prevented from moving laterally in order for the apparatus to operate properly.

In the embodiment of Figure 2, vertical deflections in the coupling members 14a and 14d will also result in a relative lateral movement of the probe in a direction

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illustrated as across the page. This is accounted for in the preferred embodiment by the compensating effect of deflections in the leaf-springs 14c and 14b which, through interaction with an intermediate member 21 and leaf springs 14a and 14d assure that no "across the page" lateral movement of the probe 12 occurs. Leaf springs 14c and 14b are selected such they shorten laterally by precisely the same amount as springs 14a and 14d in response to a given vertical displacement of the probe 12 relative to the carriage 15. In the simplest case this can be achieved by making springs 14a, 14b, 14c and 14d of identical stiffness and length. In the embodiment of Figure 2, the first and second measuring means 16, 17 take the form of non-contacting displacement measuring devices. There are cores 16a, 17a embedded within the probe 12 which, in this case, must be made from a light, non-magnetic material, such as, for example, titanium aluminium alloy or hyrilium. Magnetic cores 16a, 17a form the moving elements of linear variable differential transformers (L.V.D.T.) however other forms of inductive measuring devices could be used. This arrangement provides virtually infinite resolution of measurements. Alternatively, one or both of the measuring apparatus may take the form of a capacitance measuring device.

It should be noted that because the first measuring means 16 measures the movement of the probe with respect to a datum, it provides an output including information regarding the amount of penetration of the indenter 20 into a sample. As the non moving part 17b of the second measuring device 17 is rigidly fixed to the carriage 15, an output indicating movement of the probe 12 relative to the carriage 15 is provided. Thus, deflection of the springs 14a, 14b, 14c and 14d is measured and the actual load acting on the indenter can be precisely calculated. Furthermore, it should be realised that the arrangement of

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offsetting the probe 12 with respect to the drive carriage 15 is non-essential. A concentrically arranged embodiment is described hereinafter but the offset arrangement of the embodiment of Fig. 2 has been found to provide ease in calibration.

In use, a sample (not shown) is mounted on the surface 13 of the stage 11 beneath the indenter 20. The actuator drives the carriage 15 downward and, in turn, the probe 12 is also carried downwards. The motion of the probe 12 and the carriage 15 are the same until the indenter 20 contacts the sample surface and the springs 14a, 14b, 14c and 14d deflect. This deflection is identical to the relative movement between the probe 12 and carriage 15 and, it is therefore, precisely measured by the second measuring means 17. Consequently, the instant of contact between the indenter 20 and the sample can be ascertained and the force acting on the probe can be precisely calculated. Any relative movement of the probe 12 measured by the first measuring means 17 after the moment of contact detected by the second measuring means 16 is indicative of penetration by the indenter 20 into the sample.

To derive loading and unloading curves, a maximum force, a force increment, a dwell and a rate of incrementation are specified. The indenter is brought into contact with the surface at the lowest force in the selected range. The indenting force is then incremented to the selected maximum value at the specified rate and then decremented at the same rate. Depth of penetration is recorded at the end of the dwell between each increment or decrement from the point of contact until the force is reduced to zero.

Loading/unloading plots may be constructed and displayed with a best fit line inserted tangent to the initial part of the unloading curve. Hardness values may

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be calculated for each force level for which the loading/unloading plots are constructed, on the basis that the tangent to the initial part of the unloading curve is independent of the depth of penetration. Alternative
5 plots of hardness versus the force increment or hardness versus depth of penetration may be displayed.

Using the present invention, it is possible to monitor total indentation, indentation velocity and the load on the indenter simultaneously. Using an appropriate
10 control system, it is also possible to continue driving the actuator 19 after contact until the predetermined test load is almost reached then reduce the indentation speed so that overshooting of the test load value does not occur.

The control system of the preferred embodiment is
15 illustrated schematically in Fig. 3. A force command signal 30 which is proportional to a desired load is generated by a computer 31 is relayed to a command unit 32, which takes the form of a linear analogue ramp generator. The slope of the ramp is used to control the
20 indentation speed of the actuator 19. In order for the indentation speed to be controlled, information from the first measuring device 16 is continually compared with the ramp slope an error signal is generated and the current or voltage from the driver 24 to the actuator 19 is varied
25 accordingly. Thus, indenter velocity and displacement can be accurately controlled even though the probe 12 and the carriage 15 of Fig. 2 are not rigidly fixed together.

The output of the second measuring means 17 is compared with the force command signal 30 an error signal
30 is generated to vary the slope of the command ramp as the measured force approaches the same value as the force command signal 30. The slope of the ramp may be reduced to zero when the measured load signal is the same as the command load signal or reversed when the measured load
35 signal exceeds the force command signal.

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Furthermore, contact detector 40 generates contact signal 41 in response to the generation of a positive force by the second measuring device 17 and the command unit 37 generates a full force signal 42 when the measured force signal is the same value as the force command signal. The position signal 50 and force signal 51 are continuously recorded by the computer 31 and control lines 52 exist between the computer 31 and the command unit 37 to allow initialisation of the system.

10. A suitable computer programme is shown in skeleton form in the flow chart of Fig. 4. It comprises a series of loops controlling the four main phases of the indentation process and is considered to be self-explanatory.

15 Finally, a second embodiment of the present invention is illustrated in Fig. 5. This embodiment comprises stage 70 for mounting a sample thereon and a probe 71 with a micro-indenter 72 in the lower end thereof extending axially in a direction orthogonal to the planar upper surface 73 of the stage 70. The probe 71 is connected by elastic coupling means in the form of spring plate 74a, 74b to a carriage 75. The carriage 75 is in turn connected to a housing 80 by plate springs 76a, 76b. Plate springs 76a, 76b are slit at right angles to the slits in plate springs 74a, 74b, thus ensuring axial movement of the probe 71. In this embodiment, a first measuring device comprising a concentric capacitor 77 is used to measure the movement of the carriage relative to a datum and a second measuring device 78 comprising an L.V.D.T. or other inductive measuring device is used to measure movement of the probe 71 relative to the same datum. Thus, the deformation of the elastic connection means is also measured, being the difference between the measurements of the first and second measuring devices 77 and 78.

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Also it is possible to perform a scratch test using the present invention. A force value, a length of traverse, a traverse rate and a number readings may be specified. The indenter is brought into contact with the surface at the specified force which is maintained while the specimen is traversed through the specified distance at the specified rate. The depth of penetration is measured at the specified number of intervals and recorded. The embodiment of Fig. 2 is capable of applying forces in 3 ranges:

From the foregoing it will be clear to a skilled addressee that apparatus may take many forms without departing from the scope or spirit of the present invention.

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CLAIMS :

1. A penetration measuring apparatus comprising:
a stage for mounting a sample of a material to be tested thereon;

a probe having one end thereof proximal to said stage and having a longitudinal axis extending orthogonally of said stage;

an ultra-micro hardness indenter located in said one end of said probe;

a carriage assembly to which said probe is connected by elastic connection means of known stiffness, said carriage assembly and connection means allowing axial movement of said probe but preventing movement of said probe in any other direction;

means to drive said carriage towards and away from said stage;

means to measure the movement of said probe relative to a stationary datum; and

means to measure the deformation of said elastic connection means connecting said probe to said carriage.

2. Apparatus according to claim 1 in which said probe is made from a light non magnetic material.

3. Apparatus according to claim 2 in which at least one of said measuring means is a linear variable differential transformer having a moveable magnetic core embedded in said probe.

4. Apparatus according to claim 1 in which said elastic coupling means between said probe and said carriage comprises;

at least two leaf springs of known stiffness connected to said probe and extending laterally therefrom to an intermediate member which is axially parallel to said probe;

at least two leaf springs of known stiffness connected to said intermediate member and extending

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laterally therefrom to said carriage; axial movement of said probe causing deflections and corresponding shortening of said leaf springs wherein said shortening is identical for all leaf springs.

5. Apparatus according to claim 4 wherein said leaf springs are all of identical stiffness and length.

6. Apparatus according to claim 1 in which said elastic connection means comprises at least two spring plates.

7. Apparatus according to claim 1 in which said drive means comprises a linear electromagnetic actuator, an electromagnetic shaker or a peizo-electric actuator.

8. Apparatus according to claim 1 including a control system further comprising a command unit in the form of an analogue ramp generator, the shape of said ramp being proportional to indentation speed;

an actuator driver;

and a servo system to compare the output of said first measuring means with the output of said command unit, to generate an error signal representative of any difference in said measuring means output and said command unit output and to vary an input signal to the driver to maintain the indenter speed indicated by said ramp slope.

9. An apparatus according to claim 8 in which said control system further comprises a servo system to compare a force signal generated by the second measuring means with a signal indicative of a test force and to provide a signal to said command unit to generate a ramp of positive gradient when said measured force signal is less than said test force signal, to generate a slope of zero gradient when said measured force signal is equal to said test force signal and to generate a ramp of negative gradient when said measured force signal is greater than said test force signal.